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A Photoluminescence Technique for Detection of Gunshot Residue

When a suspect has been apprehended following a shooting, detection of gunshot residue on his hands may provide significant evidence in the investigation. The determination of the firing distance (distance from weapon to target) by the distribution of gunshot residue around a bullet hole is also useful in various gunshot investigations. We describe here the results of a preliminary study of the application of photoluminescence techniques to gunshot residue detection. The key objective in this study was to develop a rapid, reliable, and convenient method of detection for use in the crime laboratory.

Previous methods of gunshot residue detection, which are of questionable reliability because of their lack of sensitivity or specificity, include the color test for nitrates [1] and the color tests of Harrison and Gilroy [2] for antimony (Sb), barium (Ba), and lead (Pb), the three most characteristic metallic elements found in gunshot residue. Until recently, the method in general use for detecting gunshot residue on hands, although the use of this method was not nearly as widespread as need would dictate, was the application of neutron activation analysis to detect Sb and Ba [3]. This method has serious drawbacks, for example, the time and inconvenience of sending samples out for analysis and the inability to detect Pb.

Other analytical techniques being considered for detection of gunshot residue include atomic absorption spectrometry, atomic fluorescence spectrometry, charged-particle-induced and X-ray-induced X-ray emission, electrodeless plasma emission spectrometry, and molecular photoluminescence spectrometry. The cost, analysis time, and sensitivity of these techniques differ widely. General interest has been primarily in atomic absorption spectrometry. Flame atomization can be used for Pb detection [4], but flameless systems such as the graphite furnace [5] and the microthermal atomizer are required for Sb and Pb detection. One disadvantage in the use of atomic absorption is the requirement that a separate excitation lamp be installed for each element to be detected. The use of atomic fluorescence spectrometry can potentially provide multielement analysis and greater sensitivity, but research in developing the analytical technique is required before its usefulness in the detection of gunshot residue can be assessed.

Charged-particle-induced X-ray emission is sensitive enough for the detection of all elements in gunshot residue [6], but the instrumental system is expensive and not readily available in the crime laboratory. In previous studies, *a*-particles, rather than electrons, were used to induce X-ray emission. The use of heavier particles increases sensitivity

because background radiation is smaller. It would appear that a scanning electron beam such as in an electron microprobe or in a scanning electron microscope would be adequately sensitive to detect elements characteristic of gunshot residue and to correlate their presence with specific particles, but these instruments are costly. Energy dispersive X-ray fluorescence [7] appears to be sensitive enough for detecting Pb in gunshot residue and the equipment is less costly, but this method probably is not sensitive enough to detect Sb and Ba in hand washings.

Electrodeless plasma emission spectrometry has only recently been used in the detection of gunshot residue [8]. This technique has exciting possibilities because it has extremely high sensitivity and would only require attachments for spectrographs already found in some crime laboratories.

In our study we concentrated on the development of a molecular photoluminescence technique to detect metallic elements in gunshot residue. As discussed subsequently, the use of this technique is attractive because of the ease of analysis and its sensitivity and low cost. In this preliminary study we were concerned with the detection of Pb, Sb, and Ba; we did not attempt to repeat the extensive work already carried out with neutron activation analysis concerning the importance of the detection of these elements and the interpretation of findings.

Description of Photoluminescence Techniques

Background

For our purposes, photoluminescence can be defined as the light emitted by a chemical species in the ultraviolet-visible wavelength region of the electromagnetic spectrum (300 to 700 nm) when excited with ultraviolet radiation (190 to 380 nm). Absorption of ultraviolet radiation by a luminescent molecule causes it to undergo an electronic transition from the ground state, that is, the state of lowest energy, to a higher energy or excited state. When a molecule in the excited state returns to its ground energy state, a portion of its excess energy is released through the emission of light. Luminescent properties of use are the excitation[2] and emission spectra (that is, intensity versus wavelength), the decay time of the luminescence once the excitation source is extinguished, and the quantum yield of emission (that is, the ratio of the number of molecules that emit light to the number of molecules that absorb excitation). The luminescence can consist of both fluorescence and phosphorescence. The fluorescence of most molecules appears at shorter wavelengths and has a fast decay time (10^{-9} to 10^{-6} s), whereas the phosphorescence appears at longer wavelengths and has a longer decay time (10^{-6} to 10 s).

Photoluminescence analysis has the advantages that (1) it can be highly selective because the absorption, emission, and lifetime parameters must match; (2) it is highly sensitive; (3) it is nondestructive; (4) it is inexpensive; and (5) the separation of complex mixtures is unnecessary. The most beneficial attribute is the high sensitivity, which is less than a nanogram for efficient emitters. Forensic toxicologists are already using this sensitivity in screening for morphine in body fluids by means of photoluminescence [9].

Detection of Sb, Ba, and Pb

We have surveyed the literature concerning photoluminescence for methods of analysis for Sb, Ba, and Pb that would (1) be reliable, sensitive, and quantitative; (2) not involve

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[2] The excitation spectrum is a plot of the variation in the luminescence intensity as the wavelength of the exciting radiation is varied.

a great deal of wet chemistry; and (3) be capable of simultaneous determination of more than one of the three elements. The luminescent reactions of Pb(II) with morin (5.0-μg/ml sensitivity) [10] and with lucigenin (0.4-μg sensitivity) [11] do not provide the required sensitivity for determination of Pb in hand washings. The luminescent reactions of Sb(III) with Rhodamine 6G (0.1-μg/ml sensitivity) [12] and with benzoin (0.04-μg/ml sensitivity) [13] do not provide good reproducibility, and, in the case of benzoin, many elements interfere. The detection limit of Sb(III) when 3,4',7-trihydroxyflavone is used [14] is sufficient (0.04 μg), but a complex wet-chemical procedure is involved in the analysis. The most useful analysis technique involves the formation of halogen ion complexes [15,16].

Strong luminescence is observed at low temperatures for submicrogram amounts of the halogen ion complexes of thallium(I), Pb(II), bismuth(III), Sb(III), tellurium(IV), selenium(IV), and arsenic(III), all of which are ions with the electronic shell configuration $1s^2 \ldots np^6 nd^{10}(n + 1)s^2$.[3] No luminescence is observed from 45 other inorganic ions under the same experimental conditions [16]. The luminescence quantum yields of the Pb(II) and Sb(III) halogen ion complexes increase tremendously with a decrease in temperature, and, at 77 K (the boiling point of liquid nitrogen), the Pb(II) and Sb(III) chloride ion complexes form clear rigid glasses in 7M HCl.

Antimony sulfide (stibnite) (a primary constituent of cartridge primers) can be dissolved in acids. In the presence of concentrated HCl, the trivalent Sb ions form a complex with the chloride ions [17]

$$Sb^{3+} + 4Cl^- \rightarrow SbCl_4^- \tag{1}$$

$$Sb^{3+} + 6Cl^- \rightarrow SbCl_6^{3-} \tag{2}$$

and these complexes are responsible for the molecular luminescence. When lead styphnate (another primary constituent of bullet primers) is added to concentrated HCl, luminescent Pb chloride ion complexes are readily formed [17].

$$Pb^{2+} + 4Cl^- \rightarrow PbCl_4^{2-} \tag{3}$$

Barium (from the barium nitrate in cartridge primers) does not luminesce in the frozen HCl solutions.

Low temperature chloride ion complexing with Pb(II) and Sb(III) provides the most sensitive, convenient, and rapid method of luminescence analysis known for these ions; it also provides the capability of simultaneously analyzing for both ions. As shown in Figs. 1 and 2, the emission spectrum for Pb(II) peaks at 390 nm, and for Sb(III) the emission peaks at 620 nm. The band peaking at 425 nm (Fig. 2) is a combination of scattered light and HCl impurity emission. The excitation spectra (Figs. 1 and 2) peak at 276 nm for Pb(II) and at 250 nm and 300 nm for Sb(III). These emission spectra have been corrected for the variation with wavelength of the response of our photomultiplier and grating. The excitation spectra have not been corrected for the variation in the lamp intensity versus wavelength. Thus, the excitation maxima can differ for different lamps. For Pb, however, the band is so sharp that no dependence upon the lamp is expected (if we assume that the spectral output of the source does not vary rapidly with wavelength).

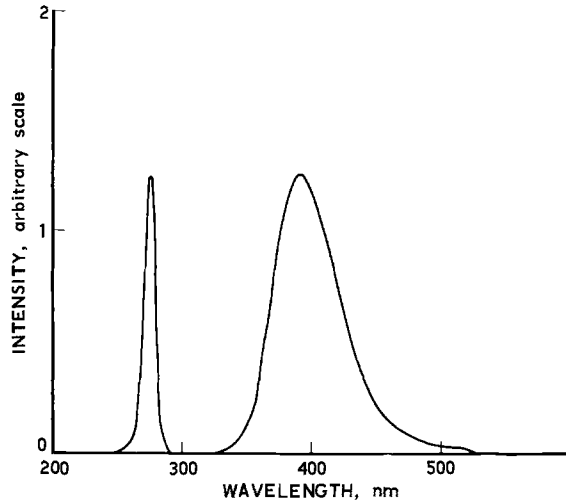[3]The luminescence is analogous to the intense $^3P_1 \rightarrow {}^1S_0$ emission from mercury atoms at 253.7 nm.

FIG. 1—*Excitation spectrum (at 390-nm emission) and corrected emission spectrum at 276-nm excitation for 1.0 μg/ml Pb(II) in 7M HCl at 77 K.*

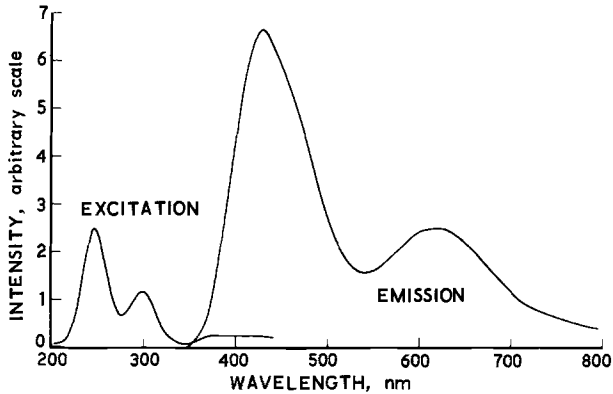FIG. 2—*Excitation spectrum (at 620-nm emission) and corrected emission spectrum (250-nm excitation) for 0.20 μg/ml Sb(III) in 7M HCl at 77 K. The emission peak at 620 nm is a result of Sb, and the peak at 425 nm is a combination of scattered light and HCl impurity emission.*

Detection of Pb and Sb in Gunshot Residue

Our primary objective in the development of this method of gunshot residue detection was to efficiently remove and collect, in a form suitable for photoluminescence analysis, all gunshot residue from a predetermined area of a hand. We were also concerned with efficiently forming luminescent chloride ion complexes with the Pb and Sb present in the collected residue.

Sample Collection

After a shooting, the first step, currently, is to wash both hands of the person who shot the gun with a stream of distilled water from a polyethylene squeeze bottle and to collect the hand washings in a large beaker. Kilty [18] recently reported that a stream of water such as this can remove over 80% of the residue. A small clean brush had been used in conjunction with the stream of water, but the method was abandoned because of the possible luminescent contamination from the brush itself and the observations that gunshot residue particles were caught in the bristles of the brush and large quantities of epithelial tissue were removed by the brush. The luminescence background caused by the large quantity of epithelial tissue was decreased when the brush was not used and centrifugation of the sample prior to analysis was no longer required. In removal of the residue from the hands following a shooting, the stream of distilled water is applied only to selected areas of the hand, namely the back of the hand, the thumb-web area, and the back of the fingers. The inside areas of the fingers and palm are avoided in the washing because of the likely placement of residue on the inside of the hand resulting from the handling of an unclean weapon.

Preliminary examination of the use of a cotton swab for removal of gunshot residue from hands indicated that strong, long-lived luminescence background is introduced by the cotton, making this method of removal undesirable in conjunction with luminescence analysis. Preliminary examination of the use of 1.2% HNO_3 in removal of gunshot residue from hands indicated that the HNO_3 completely quenches the luminescence of Sb(III) and reduces the luminescence of Pb(II).

Analysis of Hand Washings

The collected residue particulates are separated from the hand washing by filtration through clear plastic membrane filters of 5.0-μm pore size. Figure 3 is a typical photograph of a filter from a two-round firing after filtration of the collected hand washing. One millilitre of 7M HCl is used to remove the residue from the membrane filter and form the luminescent chloride ion complexes of Sb and Pb. This HCl molarity ensures a maximum luminescence intensity (maximum quantum yield of emission) for the Sb and Pb complexes and a clear rigid glass when the solution is cooled to 77 K. We have also investigated the use of HBr as a solvent, but use was abandoned because of Pb impurity levels found in the HBr and interferences in the Sb determinations as a result of other luminescent impurities. The 1-ml solution of acid residue is placed in a Suprasil® quartz sample tube of precision bore 4-mm inside diameter and 6-mm outside diameter, and the tube is directly immersed in liquid nitrogen in a quartz optical Dewar flask. The sample is then ready for ultraviolet excitation and the recording of luminescence or excitation spectra for Sb and Pb determinations. The total sample collection and analysis time should be less than 30 min.

One acid residue sample was heated in an attempt to maximize the dissolution of the residue particles. Heating the sample at 81°C for 10 min had no effect on the Pb analysis, but the luminescence from Sb was completely eliminated. A similar result was observed in the heating of a sample of fired gunshot primers in 7M HCl. However, heating of antimony potassium tartrate in HCl had no effect on the luminescence intensity.

The concentrations of Sb and Pb in the sample tubes are determined from calibration plots of luminescence intensity versus concentration for standard solutions of Sb and Pb salts. The calibration curves for our system are shown in Fig. 4. Indistinguishable curves
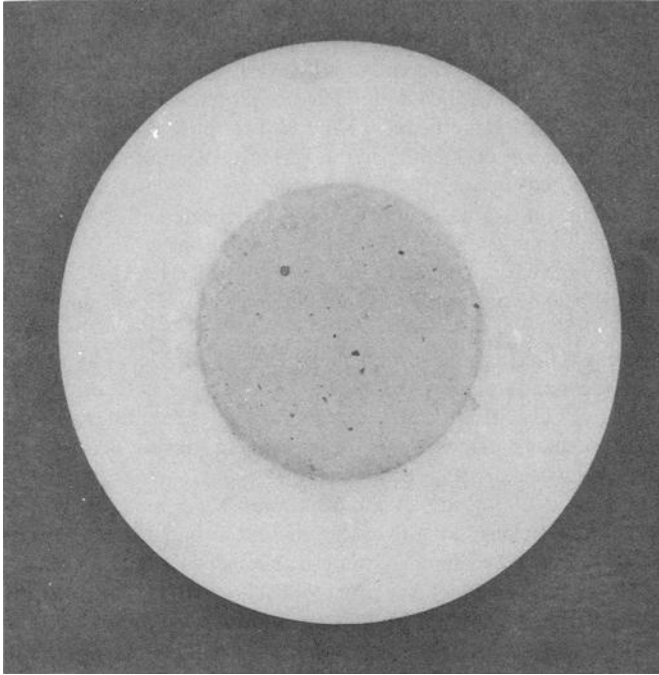
FIG. 3—*Twenty-five-millimetre-diameter membrane filter and holder, following a two-round firing and subsequent filtration of the collected hand washing.*
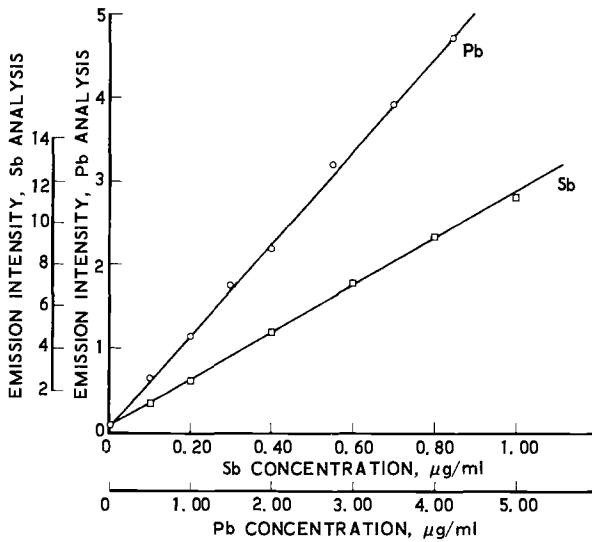

FIG. 4—*Photoluminescence calibration plots for Sb(III) and Pb(II) in HCl at 77 K. Excitation and emission wavelengths are 250 nm and 620 nm, respectively, for Sb(III), and 276 nm and 390 nm, respectively, for Pb(II).*

are obtained by adding known concentrations of Sb to a hand washing from a person who has not fired a gun. These curves change with time, for example, as a result of reduced output of the excitation source; but, because of the linear relation between intensity and concentration, it is only necessary to run one standard concentration with each set of unknowns. At higher concentrations these curves become nonlinear. For completeness a blank should also be run. Our detection limits are 0.1 and 1.0 ng for Pb and Sb, respectively, in 1.0 ml of $7M$ HCl. A strong emission from the hand-washing peak at 425 nm limits our useful detection sensitivity for Sb to 10 ng, but the Pb analysis is not hampered by this background.

Following the photoluminescence analysis at 77 K, the quartz sample tube must be rapidly heated to prevent breaking and loss of the tube as a result of the difference in the coefficients of expansion of the frozen acid and the quartz.

Typical data for Pb and Sb determinations are shown in Figs. 5 and 6 for three hand-washing samples received as unknowns. In the analysis, one person had fired two
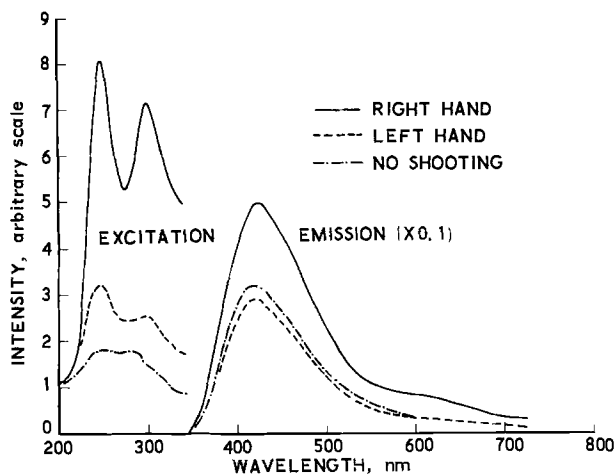

FIG. 5—*Analysis of three hand-washing samples, received as unknowns, for Sb. The solid line and broken line spectra refer to the right and left hand washings, respectively, from a person who had fired two rounds from a .380 Browning automatic pistol with his right hand. The dashed-dotted line spectrum is from the right hand of a second person at the scene of the shooting, who did not fire a weapon. The solid, broken, and dashed-dotted line spectra indicate 0.18 µg, 0.03 µg, and no detectable Sb, respectively.*

rounds, holding the gun in his right hand, while a second person stood close by. Analysis of the hand washing from the right hand of the person who shot the gun yielded 0.60 µg Pb and 0.18 µg Sb. A hand washing of the second person's right hand (he did not fire the gun) yielded 0.30 µg Pb and no detectable Sb.

Twenty-nine pairs of right-left hand washings from numerous persons, following two-round test firings of a .380 Browning automatic pistol, were taken at an outdoor shooting range. The persons' hands were always washed before handling the gun. Analysis of these hand washings for Sb after firing, showed that 20 out of the 29 firing (right) hands yielded greater than 0.04 µg Sb, whereas only 2 of the 29 nonfiring (left) hands yielded greater than 0.04 µg Sb. Figure 7 shows that amounts ranging from in-
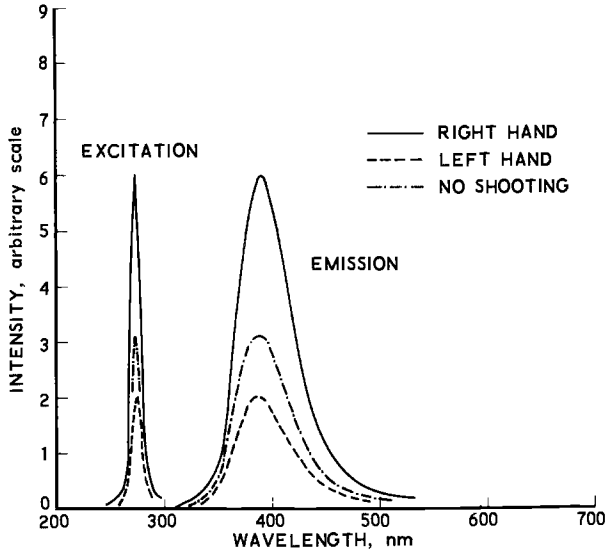
FIG. 6—*Analysis of the three hand washings for Pb. The three samples were the same unknowns analyzed for Sb in Fig. 5. Analysis of the right hand (shooting hand) of the person who fired the gun yielded 0.60 µg Pb.*
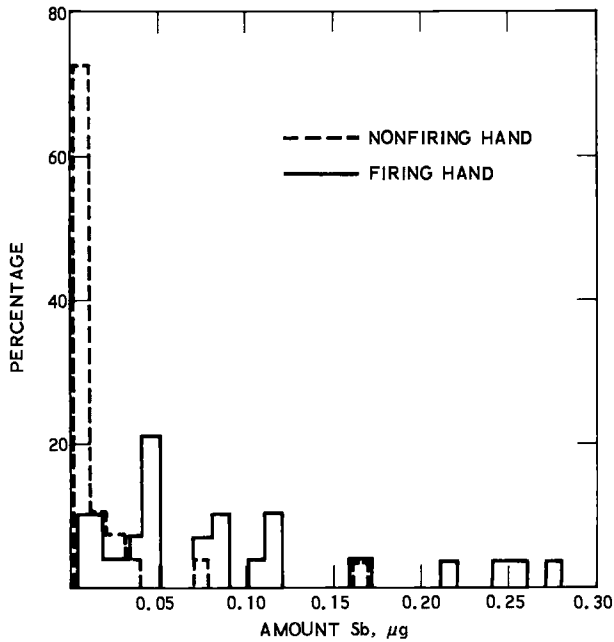

FIG. 7—*Antimony found in the hand-washing samples from firing and nonfiring hands following two-round firing of Browning .380 automatic pistol.*

distinguishable levels (<0.005 μg) to 0.27 μg were observed for Sb on the firing hand during these test firings. Calculation of the ratio of Sb amounts found on the firing (right) hand to nonfiring (left) hand shows that 85% of the samples gave a ratio greater than 4.0 and 60% of the samples gave a ratio greater than 50. However, on three occasions, the ratio was less than 1. Out of 20 hand blanks taken from persons while in the laboratory, no Sb was detected. For hand blanks taken at the pistol range before shooting, traces of Sb were observed, but with an upper limit of 0.01 μg Sb. This background level seems to present no serious problem for determining whether or not a person has fired a gun because Sb levels in the 0.04 to 0.12-μg range are commonly observed on firing hands after a shooting.

Analysis of the 29 pairs of hand washings for Pb, following the two-round firings, gave a range from 0.10 to 2.9 μg for amounts of Pb found on the firing hand, and 80% of these yielded more than 0.60 μg of Pb. Amounts of Pb detected in 20 hand blanks taken from persons while in the laboratory gave a range from 0.10 to 0.60 μg (Fig. 8). Ratios calculated for amounts of Pb found on the firing hand to the nonfiring hand ranged from 0.35 to 11. For the hand blanks collected in the laboratory, the ratio of the amounts of Pb on the right hand to left hand ranged from 1.1 to 2.6.

We observe that repeated washings of the same hand (with a stream of distilled water) lowers the background Pb found on the hand. Therefore, our Pb analyses for the firing hands, which were always washed prior to test firings, will lack the 0.05 to 0.20 μg of background Pb. For hand blanks taken at the target range before test firing, the amount of Pb differs from that for the hand blanks taken in the laboratory. The amounts of Pb
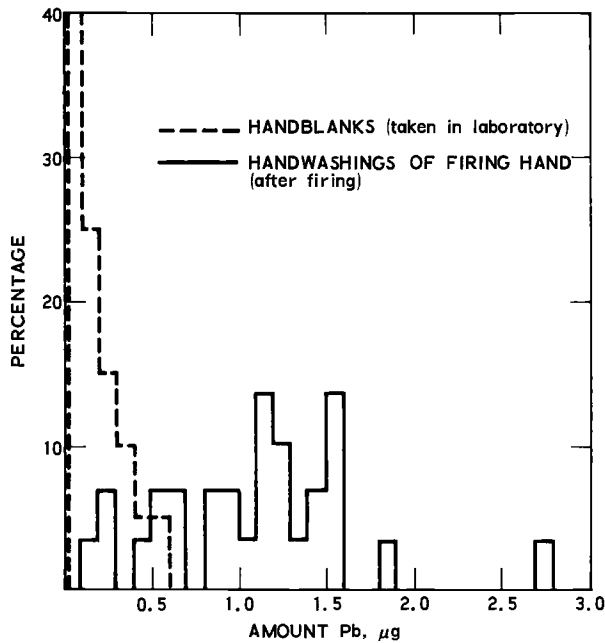

FIG. 8—*Lead found in the hand blanks taken in laboratory and hand-washing samples from firing hand following two-round firings of Browning .380 automatic pistol.*

seen in hand blanks taken at the firing range varied from 0.10 to 1.13 μg for 16 samples.[4]

Identification of Gunshot Residue on Cloth

In a preliminary examination of the application of the photoluminescence technique for identification of bullet holes in cloth and for potential use in determining firing distance, one round was fired from a .380 Browning automatic pistol through a piece of an old cotton cloth at a distance of 2 ft from the end of the pistol barrel. A 1.5-cm-diameter cut was made around the bullet hole. Soaking this cut of cloth in 7M HCl yielded strong luminescence intensity at 77 K corresponding to 0.2 μg Sb and 1.4 μg Pb. Soaking a similar-sized piece of the same cloth (removed before the shooting) in 7M HCl yielded very weak luminescence corresponding to 0.06 μg Pb and no detectable Sb.

Equipment Used

The luminescence analysis of the acid gunshot residue samples was carried out with a Aminco-Bowman Model 4-8202 spectrophotofluorometer, which is equipped with a 150-W xenon d-c arc lamp as excitation source. The sample luminescence was detected with an RCA Model C31034 photomultiplier. A Hamamatsu Model R136 photomultiplier was also used, but the RCA model was favored because of its higher sensitivity in the red region of the electromagnetic spectrum and because of its flat spectral response from 400 to 700 nm. A Keithley Model 417 picoammeter was used to amplify the signal, and the spectra were recorded with a Moseley Model 2D X-Y recorder. An Aminco Model B28-62140 micro Dewar flask was used for liquid nitrogen immersion of the quartz sample tubes. Nucleopore membrane filters were 25 mm in diameter with 5.0-μm pore size. The HCl was Mallinckrodt Chemical Works reagent grade; the antimony potassium tartrate, for standard solutions of Sb(III), was J. T. Baker Chemical Co. USP grade; and the lead nitrate, for standard solutions of Pb(II), was Baker and Adamson quality label reagent grade.

A filter fluorometer conceivably could be used instead of a spectrophotofluorometer to control the wavelengths of luminescence excitation and emission for detection of Sb and Pb in gunshot residue. The filter fluorometer is an uncomplicated, less expensive system and would do the job just as well as the spectrophotofluorometer.

Conclusions and Discussion

This preliminary investigation of the application of the photoluminescence technique for the detection of gunshot residue has established a rapid, convenient, sensitive, and quantitative method for the detection of Pb and Sb on the hands. Based upon the *limited* number of samples for the single firearm studied to date, ratios of the amounts of Pb and Sb found on the firing hand to the nonfiring hand and the absolute amounts of these elements can be used to determine that a person might have fired a gun. We expect our method of residue collection to have lower background levels than those of some other methods because our method analyzes only particulates not readily soluble in water. A cotton swab moistened with HNO_3, for example, will remove material from the hands other than particulates.

Our method exhibits potential as a screening procedure that could be used immediately following the apprehension of a suspect. A portion of the filter containing hand-

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[4] One possible source of Pb and Sb contamination prior to test-firing is the presence of gunshot residue in the air, because other guns were being fired in the vicinity of our experiment.

washing residue could be rapidly and simultaneously analyzed for the presence of Sb and Pb by means of our photoluminescence method as a presumptive test for the presence of gunshot residue. The residue on the remaining portion of the filter might then be examined in greater detail later by another, more definitive technique or reserved as a "retain" sample. Our work also shows the possibility of applying photoluminescence analysis for Sb and Pb to the identification of bullet holes and the determination of firing distance.

Considerably more experimentation is planned to increase the utility of photoluminescence analysis and better define its limitations. A more convenient method of removing the residue from suspects' hands is of particular concern. It should be possible to use cotton swabs such as used for other analytical procedures, but a chemical separation of the element, as is currently recommended for flameless atomic absorption analysis, may be required. The examination of many calibers and types of ammunition and guns will be pursued, as well as a thorough investigation of the common levels of Pb and Sb found on the hands when no gun has been fired. Photoluminescence methods to analyze for Ba and copper (Cu) in gunshot residue will also be investigated.

Summary

Rapid, convenient detection of gunshot residue on the hands of a suspect, following a shooting, can be accomplished by the photoluminescence determination of the presence of lead (Pb) and antimony (Sb), which originate in the cartridge primer. Following the firing of a gun, the backs of both hands are washed in a stream of distilled water. Each hand washing is filtered, and the residue collected on a membrane filter is dissolved in hydrochloric acid. Lead(II) and Sb(III) form chloride ion complexes with the acid, which luminesce strongly upon selective ultraviolet excitation at low temperature. Upon excitation, the Pb and Sb complexes emit light with maxima at wavelengths characteristic for the two metallic elements. By the use of this procedure, it is possible to detect as little as 1.0 ng of Pb and 10 ng of Sb. The total time for sample collection and analysis is less than 30 min.

Acknowledgments

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